International Journal of Metallurgical &
Materials Science and Engineering (IJMMSE)
ISSN(P): 2278-2516; ISSN(E): 2278-2524
Vol. 7, Issue 2, Apr 2017, 1-6

TRANS STELLAR
•Journal Publications •Research Consultancy

STRIKING ENHANCEMENT OF MAGNETIC SOFTNESS IN SHORT TIME ANNEALING THAN FOR LONGER ANNEALING TIME OF $Fe_{74}Cu_{0.8}Nb_{2.7}Si_{15.5}B_7$

UMASREE DHAR[1], SHEIKH MANJURA HOQUE[2] & DEB PRASAD PAUL[3]

[1]*Assistant Professor, Bangladesh Open University, Gzipur, Bangladesh*

[2]*Material Science Division, Atomic Energy Centre, Dhaka, Bangladesh*

[3]*Deptartment of Physics, Chittagong University, Chittagong, Bangladesh*

ABSTRACT

A thorough study has been performed on FINEMET type of ribbons with nominal composition of $Fe_{74}Cu_{0.8}Nb_{2.7}Si_{15.5}B_7$ synthesized by rapid solidification technique. From XRD, it was observed that the as-cast sample was completely amorphous. In order to correlate microstructural feature with soft magnetic properties, grain size, Si content, lattice parameter were determined for various time and temperatures. The soft magnetic properties of this nanocrystalline alloy annealed at 500-650°C for 1 to 60 minutes at regular interval are investigated. It was striking to note from the experiment is that optimum value of permeability relevant to a definite temperature is attained within few minutes for example 1-5 minutes. Further annealing does not lead to significant enhancement of permeability. In our experiment, highest value of permeability of about 43000 has been obtained at 580°C for 3 minutes. Further increase of annealing time at a definite annealing temperature decreases the permeability value. Thus, except for the sample annealed at 550°C where monotonic increase of permeability takes place with the increase of annealing time, short time annealing within 1-5 minutes at relatively higher annealing temperature provide better soft magnetic properties than long time annealing. Change of initial permeability is governed by the grain size, composition of nanograin and residual amorphous matrix, which determines average anisotropy of the material. Also, long time effect has the effect of induced anisotropy, which reduces the value of μ´significantly. When annealing time is increased successively for the sample it is found that the induced anisotropy behaviors of the sample has become supreme and along with that decrease the permeability significantly.

KEYWORDS: FINEMET, Nanocrystalline Alloy, Permeability & Amorphous

Received: Jan 05, 2017; **Accepted:** Feb 08, 2017; **Published:** Feb 14, 2017; **Paper Id.:** IJMMSEAPR20171

Original Article

INTRODUCTION

In 1988, Yosizawa and his co-workers [l] reported for the first time on a new class of iron-based alloys exhibiting superior soft magnetic behavior. The nanocrystalline state have been attained when the sample annealed for the temperature typically from about 500°C to 600°C which also provide primary crystallization of b.c.c. Fe for the sample. As a result, the randomly oriented microstructure has been found. Also ultrafine grains of b.c.c. Fe-Si (20 at %) with grain sizes about 10-15 nm have been discovered upon the residual amorphous matrix which fill the volume of about 20% to 30% and the distance between the crystallites is about 1-2 nm. The higher values of initial permeability and lower value of coercivity represent the excellent soft magnetic properties[ll]. The highest value of initial permeability of about 10^5 and correspondingly the lower value of coercivities less than 1 A/m have been measured. Further annealing the sample at higher temperatures above 600°C, there is slight precipitation of boride compounds like Fe_2B or Fe_3B which dimensions are about 50nm to 100 nm. The particular nanocrysalline

structure which have been established, is related to the joined effect of Cu and Nb and they are low soluble in b.c.c. Fe-Si: Cu enhances the nucleation of the b.c.c. grains while Nb impedes the gain coarsening and at the same time inhibits the formation of boride compounds.

The aim of the paper is to find the optimum conditions of thermal annealing treatment leading to the increase of initial permeability and also decrease of coercive force.

EXPERMENTAL

The amorphous $Fe_{74}Cu_{0.8}Nb_{2.7}Si_{15.5}B_7$ alloy was prepared by rapid solidification of the melt by using the single roller copper wheel melt spinning technique in the form of ribbon. The ribbons were 6mm wide and 20-50 µm thick. The amorphous state was confirmed by x-ray diffraction and thermal analysis. X-ray diffraction patterns of the samples were recorded by means of Philips X'pert Pro X-ray diffrectometer at room temperature with CuKα radiation. The measurement of frequency dependence of complex part of initial permeability of the amorphous and the sample annealed at different temperatures were taken in the frequency range of 1 kHz to 13 MHz.

RESULTS AND DISCUSSIONS

The X-ray diffractogram of the sample $Fe_{74}Cu_{0.8}Nb_{2.7}Si_{15.5}B_7$ annealed at temperature 580°C for 1-10 minutes at regular interval is presented in Figure 1. It is shown from the figure that no crystalline phase has formed due to rapid quenching for as-cast sample. The evolutions of crystalline phase appears with the annealing time of 1 minute and onward at 580°C and have been identified as bcc Fe(Si) using standard software. As the annealing time increases, the peaks become sharper with higher intensity.

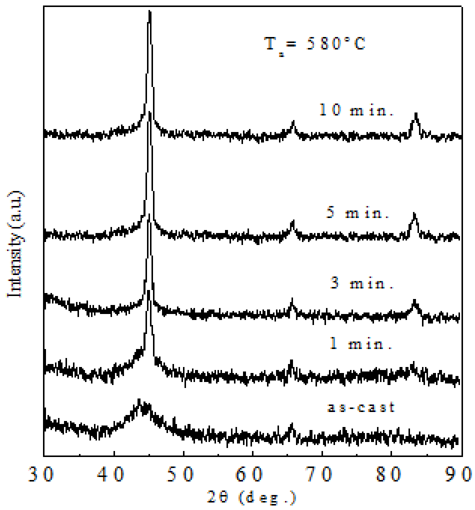

Figure 1: XRD Pattern for different Annealing Time Annealed at 580^{o}C

In Figure 2, the lattice parameters and Si content of α -Fe(Si) nanograins dispersed in the surrounding amorphous matrix have been presented for different annealing time 1 to 60 minutes at regular interval at temperature 580°C. The value of lattice parameter of pure Fe is higher than that of lattice parameter of α-Fe(Si) phases and the value is about 2.866 Å. Thus it can be said that the decrement of lattice parameter is occurred because of the compression of α-Fe lattice. As a result, the silicon with smaller atomic size is diffused into the iron lattice with larger atomic size make a substitutional solid

solution to constract α-Fe(Si) during the process of crystallization. Si content of α-Fe(Si) nanograins has been measured from the established quantitative relationship between lattice parameter and Si content of Fe-Si alloys [lll].

The change of lattice parameter follows the change of Si content. Lattice parameter decreases when Si diffuses in α-Fe(Si) lattice and again lattice parameter increases when Si diffuses out of the α-Fe(Si) lattice.

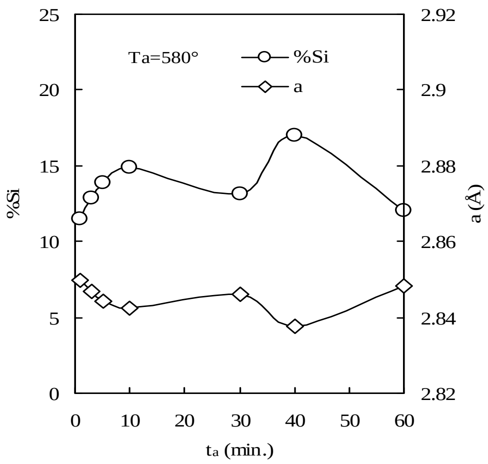

Figure 2: Variation of Lattice Parameter and Si Content at % with Annealing Time at Temperature, T_a=580°C

When the samples are annealed above certain time an increase of lattice parameter with subsequent decrease of Si content indicates that recrystallization of α-Fe(Si) grains has taken place and that during recrystallization silicon diffuses out of the α-Fe(Si) grains. Presence of higher Si content in this composition facilitates the formation of Fe_3Si phase.

Figure 3 represents the mean grain size of the bcc Fe(Si) nanograins using the Scherrer's formula at temperature T_a= 580°C for 1 to 60 minutes of annealing time. At temperature 580°C, the value of grain size is 9.35 nm for 1 minute annealing time. For higher annealing time, the limiting value attains 11.63 nm at 5 minutes and remaining constant up to 60 minutes. It may be attributed to the combined effect of Cu and Nb elements. For the annealing temperature of 580°C the variation of grain size with annealing time has changed for shorter span of time and then attains a limiting value. This shows that at higher temperature sufficient activation energy of crystallization is attained within very short time which is almost at its saturation level and does not change much even after prolonged annealing of 1 hr.

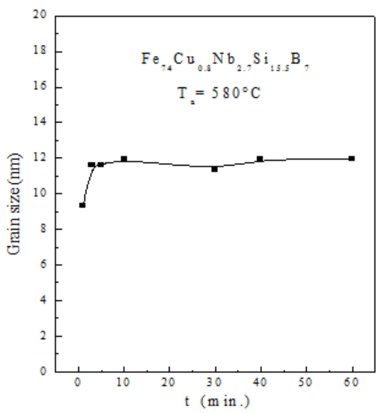

Figure 3: Annealing Time Dependence of Grain Size at 580°c

Figure 4 show μ′ as a function of frequency in the temperature range 550°C to 650°C for the fixed time 3 minute up to 1 kHz to 10 MHz. The characteristics of these curves are that μ′ remains fairly constant up to some critical frequency. At critical frequency, μ′ drops rapidly. The evolutions of nanocrystalline phase from amorphous precursor are very much time and temperature dependent. The highest permeability of about 43000 from this curve is obtained for the temperature 580°C and the lowest value is found at 550°C which is about 6000. At annealing temperature 650°C, the highest value of μ′ is reduced and it is 14000. It may be happened due to the induced anisotropy.

For higher annealing temperature or time, some of the Si diffuses out of the α-Fe(Si) lattice. In this case formation of ordered Fe_3Si phase has also been observed. The reduction of Si from α-Fe(Si) lattice and formation of Fe_3Si changes the average anisotropy of the sample as a result of which permeability decreases with higher annealing time and temperature.

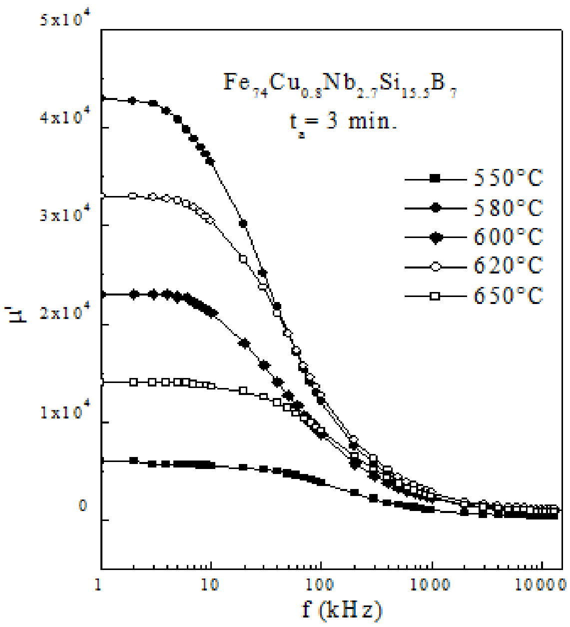

Figure 4: Variation Real Part of the Complex Initial Permeability, μ′ as a Function of Frequency for different Tem., T_a at a Fixed Annealing Time 3min

In Figure 5, the increase of the real part of complex initial permeability measured at 1 kHz has been presented as a function of cumulative annealing time for different temperature of 550°C to 650°C. For all the temperature the maximum value of μ′ is attained within 3-5 minutes. Further increase of annealing time, lowers the value of μ′. But at temperature 550°C, μ′ increases slowly. The highest value of μ′ around 43000 is attained for annealing time of 3 minutes at temperature of 580°C. As the annealing time is increased, the value of μ′ drops rapidly. Upon

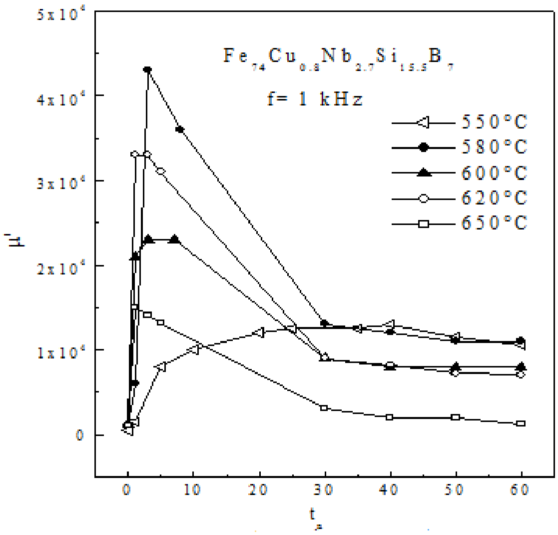

Figure 5: Variation of Initial Permeability, μ' with Annealing Time at a Fixed Frequency, f=1 KHZ

Further increase of annealing time the value of μ′ does not vary significantly up to 60 minutes annealing time. At 550°C subsequent increase of grain size led to subsequent increase of initial permeability. But variation of initial permeability has been controlled by the composition of nanograin and residual amorphous matrix which find out the average anisotropy of the material [lv].

CONCLUSIONS

The structures of $Fe_{74}Cu_{0.8}Nb_{2.7}Si_{15.5}B_7$ alloys annealed at temperature ranges of 550 - 650°C for various annealing time are investigated. It is striking that the best magnetic properties have been observed for the sample annealed at 580°C for 3 minutes and the value is 43000 which represent that short time annealing above the crystallization temperature provide better soft magnetic properties than for long time annealing. The mean grain size is of the order of 11-13 nm.

ACKNOWLEDGEMENTS

The author would like to express her heartfelt gratitude to Director, AECD and Material Science Division, AECD for their kind permission to use the laboratory of MSD, Dhaka and to the Department of Physics, University of Chittagong to instruct me to do this research work in the fascinating field of nanomaterials.

REFERENCES

1. *Y. Yoshizawa, S. Oguma, and K. Yamauchi(1988). New Fe-based soft magnetic alloys composed of ultrafine grain structure. J. Appl. Phys.64, 6044-6047.*
2. *G. Herzer. (1997). Handbook of Mag. Mater, Vol. 10, P. 427.*
3. *M. A. Hakim and S. Manjura Hoque, J. Magn. Magn. Mat. P. 395 (2004).*
4. *M. Knobel, J. P. Sinnecker, J. F. Saenger, R. Sato Turtelli(1994). J. Mag. Mag. Mater. 133, 255.*